

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	HOA et al.	Customer Number:	30448
Docket No.:	789-100	Confirmation No.:	1088
Serial No.:	10/596,750	Group Art:	1796
Filing Date:	May 15, 2007	Examiner:	Feely, Michael J.
Title:	METHOD AND SYSTEM FOR MAKING HIGH PERFORMANCE EPOXIES, AND HIGH PERFORMANCE EPOXIES OBTAINED THEREWITH		

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**DECLARATION UNDER 37 CFR §1.131**

Dear Sir:

1. We, 1) Van Suong HOA, 2) Weiping LIU, and 3) Martin PUGH, declare and state:
2. We are the co-inventors of the above-identified U.S. Patent Application entitled: "METHOD AND SYSTEM FOR MAKING HIGH PERFORMANCE EPOXIES, AND HIGH PERFORMANCE EPOXIES OBTAINED THEREWITH" (hereinafter "the application").
3. We understand that the US patent application of Drzal et al., US 2005/0119371, which claims priority on US provisional patent application 60/511,258 filed October 15, 2003, is applied as prior art against claims 9 – 16 of the application.
4. We are the same 1) Van Suong HOA, 2) Weiping LIU, and 3) Martin PUGH listed as co-inventors in the Invention Disclosure form, a copy of which is attached hereto as Exhibit A, which we prepared and submitted to Concordia University Research Office prior to October 15, 2003.

5. The Invention Disclosure Form, attached as Exhibit A, makes clear the invention claimed in the above-identified application was conceived and reduced to practice prior to October 15, 2003.
6. The undersigned further declare that the conception and reduction to practice of the invention claimed in the above-identified application occurred in Canada.
7. Exhibit A describes a need to improve the ductility of an epoxy resin matrix material. More specifically, Exhibit A states:
  - a. “Epoxy resins have been used in many applications, from aircraft composites to the ordinary household glues”<sup>1</sup>
  - b. “The function of the epoxy in these applications is to bond the fibers together to provide good load transfer by shear action across the resin matrix.”<sup>2</sup>
  - c. “Improvement in the ductility of the resin matrix material certainly can improve greatly the energy absorption capacity of the composite material.”<sup>3</sup>
8. In the context of improving the ductility of an epoxy resin matrix material, Exhibit A describes new materials, incorporating nanometer-scale clay particles, stating:
  - a. “The new materials provide fracture toughness that is many times more than the fracture toughness of the current epoxy. This increase in fracture toughness improves greatly the capability of the material to absorb energy, for example from impact, and to resist the growth of cracks.”<sup>4</sup>
  - b. “This increase in fracture toughness was obtained by the incorporation of clay particles that have dimensions in the nanometer range. The nano-scale distribution of these nanoparticles provides the enhancement in the fracture toughness of the material.”<sup>5</sup>
9. Exhibit A includes Figure 1 (reproduced below with annotations), which shows a schematic representation of a high pressure, high speed mixing process.

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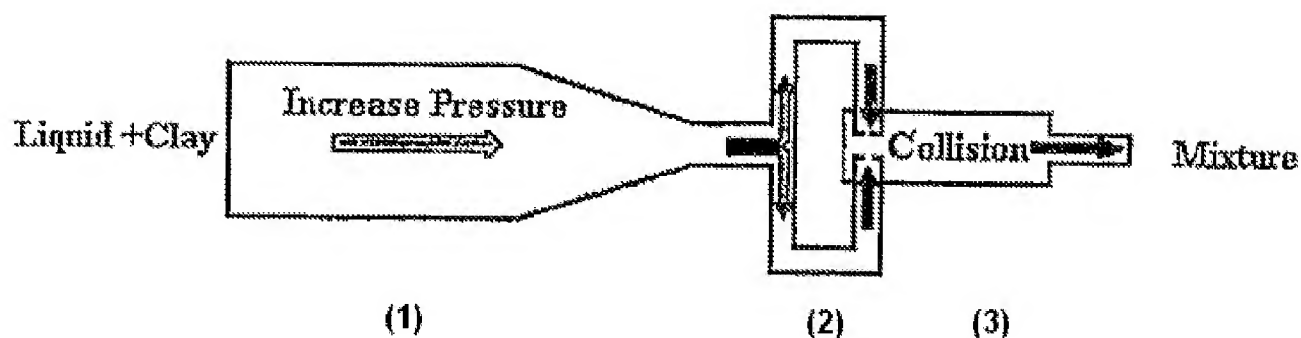
<sup>1</sup> Page 1 of IP Disclosure Form.

<sup>2</sup> Page 1 of the IP Disclosure Form.

<sup>3</sup> Page 1 of the IP Disclosure Form.

<sup>4</sup> Page 2 of the IP Disclosure Form.

<sup>5</sup> Page 2 of the IP Disclosure Form.



10. The IP Disclosure Form, including Figure 1, establishes conception and reduction to practice of the invention claimed in claim 20, as well as, claims 21 – 24 and 26, which depend from claim 20. Claim 20 is directed to a modified epoxy produced from a pristine epoxy, the modified epoxy having at least higher barrier properties and thermal resistance than the pristine epoxy. The modified epoxy according to claim 20 is produced by a process comprising steps a), b), c), and d).
11. The IP Disclosure Form supports step a) of claim 20, which recites mixing solvents and clay particles of a dimension in the nanometer range, to form a clay solution, agglomerates of clay particles forming in the clay solution. Mixing solvents and clay particles to form a clay solution, agglomerates of clay particles forming in the clay solution is disclosed throughout the IP Disclosure Form, for example, in Figure 1, which illustrates liquid and clay fed into a mixing apparatus. Furthermore, page 2 of the IP Disclosure Form describes subjecting a liquid solution to shearing to provide a nano-scale distribution of clay particles. Clay particles of a dimension in the nanometer range are also supported throughout the IP Disclosure Form, including on page 2, which explains an “increase in fracture toughness was obtained by the incorporation of clay particles that have dimensions in the nanometer range.”
12. The IP Disclosure Form supports step b) of claim 20, which recites generating a flow of clay solution. Figure 1 of the IP Disclosure Form clearly shows generating a flow of clay solution. Step b) of claim 20 further requires submitting said flow to conditions (1), (2), and (3). Each of these conditions are supported by the IP Disclosure Form attached as Exhibit A.
  - a. Condition (1) of step (b) recites submitting the flow of clay solution to high

pressure. Figure 1 of the IP Disclosure Form shows the clay solution being subjected to increased pressure in a first section, labeled (1) in the Figure above, of a mixing apparatus. Furthermore, page 2 of the IP Disclosure Form describes, “[t]he procedure used here is to run the mixture through tiny pipes under very high pressure (20,000 psi). There is significant shearing in the liquid solution when it is subjected to this flow under high pressure.”

- b. Condition (2) of step (b) recites submitting the flow of clay solution to high velocity and breaking impacts in a region of obstacles to allow the agglomerates to be broken down. A person having ordinary skill in the art would understand that Figure 1 of the IP Disclosure Form illustrates step (b). The previously-discussed first section of the mixing apparatus illustrated in Figure 1 is followed by a region of obstacles, i.e, a tortuous path having a smaller flow diameter. The region of obstacles is labeled (2) in the Figure above. The velocity of the clay solution increases as it passes through the narrower flow diameter. The region of obstacles, illustrated in Figure 1 of the IP Disclosure Form, includes splitting the flow of the clay solution into at least two separate flows. The separate flows are redirected multiple times and finally, redirected toward one another. A person having ordinary skill in the art would understand that the region of obstacles illustrated in Figure 1 of the IP Disclosure Form would allow the agglomerates of clay particles in the clay solution to be broken down.
- c. Condition (3) of step (b) recites submitting the flow of clay solution to a sudden lower pressure, yielding a dispersed clay solution having a fine and homogeneous distribution of clay particles of a dimension in the nanometer range in the clay solution. Figure 1 of the IP Disclosure Form shows that the clay solution exits the region of obstacles into a larger chamber, labeled (3) in the Figure above. A person having ordinary skill in the art would understand that the clay solution exiting the tiny pipes and entering the larger chamber would result in a sudden reduction in pressure. Furthermore, page 2 of the IP Disclosure Form describes, “[a]lso, at the end of the pipe, the material is exposed into a larger storing chamber. The sudden collapse from high

pressure in the tiny pipes to very low pressure in the chamber explodes the particles into the mist of the liquid solution of the matrix.”

13. The final Office Action mailed December 27, 2010 errs by asserting condition (3) of step (b) is not disclosed in the IP Disclosure Form. The final Office Action takes a single sentence on page 2 of the IP Disclosure Form out of context and asserts that sentences fails to provide *ipsis verbis* support for the phrase, “a sudden lower pressure, yielding a dispersed clay solution having a fine and homogeneous distribution of clay particles of a dimension in the nanometer range in the clay solution” as recited in claim 20. More specifically, in evaluating whether this phrase of claim 20 is supported in the IP Disclosure Form, the Office Action only considers the following sentence on page 2 of the IP Disclosure Form: “The sudden collapse from high pressure in the tiny pipes to very low pressure in the chamber explodes the particles into the mist of the liquid solution of the matrix.”
14. In the context of the IP Disclosure Form as a whole, a person having ordinary skill in the art would understand that the sentence upon which the final Office action focuses, can mean, “The sudden collapse from high pressure in the tiny pipes to very low pressure in the chamber explodes the particles into the mist of the liquid solution **[which can include an epoxy or which can subsequently be mixed with epoxy to form part]** of the **[ultimate epoxy]** matrix.” This interpretation is especially apparent in view of Figure 1, which illustrates a process, into which liquid and clay are fed.
15. The present application further corroborates the interpretation of the IP Disclosure Form, described above in paragraph 14. Figure 1 of the present application (reproduced below) shows a flowchart of a method for making high performance epoxies according to an embodiment of a first aspect of the present invention.

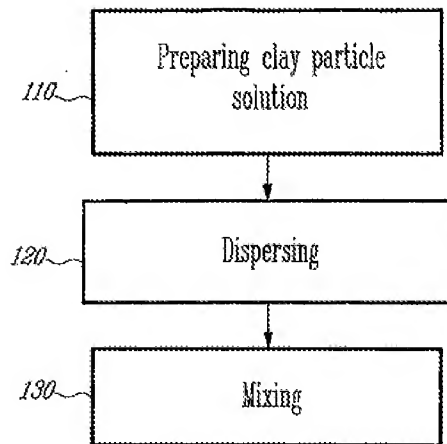


FIG. 1

Paragraphs [0053] – [0055] of the present application as filed describe Figure 1, stating “The step 110 comprises mixing solvents and clay particles of a dimension in the nanometer range in a liquid solution .... Alternatively, epoxy may also be mixed in solution with the clay particles at this state.”

16. Finally, the IP Disclosure Form supports step c) of claim 20, which recites mixing the dispersed clay solution with at least part of the pristine epoxy. This step is supported throughout the IP Disclosure Form, including page 2, which explains, “[t]he new materials provide fracture toughness that is many times more than the fracture toughness of the current epoxy. ... This increase in fracture toughness was obtained by the incorporation of clay particles that have dimension in the nanometer range.” Figure 2 of the IP Disclosure Form also shows a comparison of the fracture toughness of the developed resin as compared with other resins.
17. In accordance with common U.S. Patent Office practice on 37 CFR §1.131 Declarations (M.P.E.P. § 715.07), all indications of dates in the attached Exhibit have been deleted.
18. It is clear from the showing provided by the Exhibit attached hereto that we conceived and reduced to practice the presently claimed invention prior to October 15, 2003.
19. The undersigned further declare that all statements made herein of our own

knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application of any patent issued thereon.

Respectfully submitted,

\_\_\_\_\_  
Date

\_\_\_\_\_  
\*

\_\_\_\_\_  
Date Feb. 21, 2011

\_\_\_\_\_  
\* Li Mingzhang

\_\_\_\_\_  
Date

\_\_\_\_\_  
\*

# **EXHIBIT A**



## INTELLECTUAL PROPERTY (IP) DISCLOSURE FORM

The information on this form will be treated as confidential.

1. **Name of IP created:**

**Process for the development of epoxy nanocomposites and products thereof.**

2. **Brief description:**

This invention deals with the procedure for development of epoxy nanocomposites with outstanding properties as compared with the epoxies currently used. New epoxy materials with outstanding fracture toughness have also been invented using the procedure described.

3. **Detailed description:**

*What problem does this created IP deal with or help solve?*

Epoxy resins have been used in many applications, from aircraft composites to the ordinary household glues. The properties that allow epoxies to be used over a large range of applications are their adhesiveness, good environmental resistance and relatively good mechanical properties. Some examples are epoxies used in aircraft composite materials where the epoxy is used as a matrix to bond the reinforcement fibers together. These composite materials provide good stiffness, good strength and good fatigue resistance. The function of the epoxy in these applications is to bond the fibers together to provide good load transfer by shear action across the resin matrix. One of the weaknesses of the composite is the brittleness of the material. This is due to the brittleness of the fibers and also the brittleness of the resin matrix. Improvement in the ductility of the resin matrix material certainly can improve greatly the energy absorption capacity of the composite material. This helps in making aircraft structures more damage tolerant and thus safer. Other epoxies for commercial applications also need to have improved toughness to improve durability in operations.

*By what means has this problem be dealt with up to present?*

Up to the present time, the brittleness of the epoxy material has limited their applications. For aircraft structures, most of the composite applications are in the secondary structure. Extreme care also has been taken to ensure that the composite structure is not subjected to any impact. For other applications, the brittleness of the epoxy result in failure due to impact.

*What are the limitations or drawbacks of present methods or products?*

As mentioned above, the limitations of present materials are that they are relatively brittle. For composites, this limits their applications to secondary structures. For other applications, this may result in poorer durability.

*What capabilities of your creation overcome such limitations? And how?*

The new materials provide fracture toughness that is many times more than the fracture toughness of the current epoxy. This increase in fracture toughness improves greatly the capability of the material to absorb energy, for example from impact, and to resist the growth of cracks..

This increase in fracture toughness was obtained by the incorporation of clay particles that have dimensions in the nanometer range. The nano-scale distribution of these nanoparticles provides the enhancement in the fracture toughness of the material.

*Identify particular aspects of your creation that you believe to be original:*

The particular aspects of the creation that are original are in the mixing procedure of the material. In the normal mixing procedure, mechanical mixing is used. Mechanical mixing can only provide some limited degree of distribution of the particles down to the micro dimensions. Micro-scale distribution of the particles can only provide improvement in the order of about 20% or 30% of the fracture toughness. The procedure used here is to run the mixture through tiny pipes under very high pressure (20,000 psi). There is significant shearing in the liquid solution when it is subjected to this flow under high pressure. Also, at the end of the pipe, the material is exposed into a large storing chamber. The sudden collapse from high pressure in the tiny pipes to very low pressure in the chamber explodes the particles into the mist of the liquid solution of the matrix. As such, an extremely fine distribution of the particles down to nano-dimensions can be obtained.

*What attractive features does your creation offer the user?*

The attractive features are:

- Epoxy resins with outstanding fracture toughness.
- Epoxy resins that have other properties similar to current resins.
- Epoxy resins that do not cost more than existing resins.
- Procedure to produce other types of thermoset resins with similarly enhanced properties.

*Attach supporting information that may help explain the ideas, such as plans, sketches, photographs, drawings, flow sheets, performance data or graphs:*

Figure 1 shows the schematic of the flow mixing process.

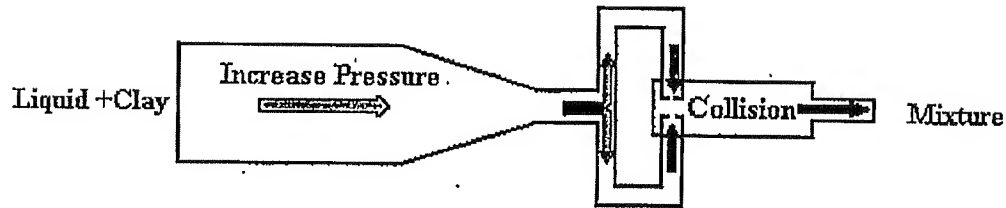


Figure 1: Schematics of the high pressure high speed mixing process.

Figure 2 shows the comparison of the fracture toughness of the developed resin as compared with current resin and also those made by the current mechanical mixing process.

## Toughness of Nanocomposites

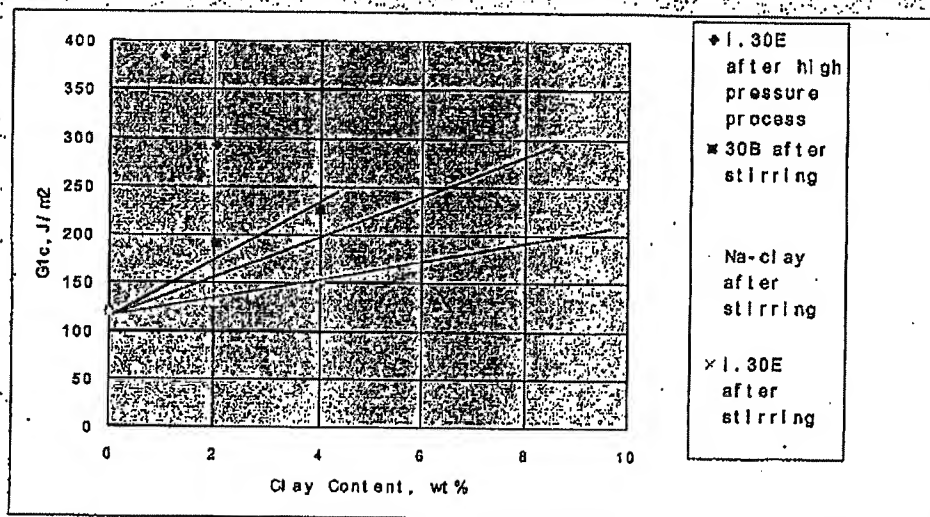


Figure 2: Comparison of the fracture toughness of one epoxy without and with clay.

4. **Creators:**

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- Dr. Martin Pugh, Department of Mechanical and Industrial Engineering, Concordia University, Montreal, Quebec, Canada, H3G 1M8, Tel. 514- 848 3170, Fax. 5140 848 3175. Email "[pugh@me.concordia.ca](mailto:pugh@me.concordia.ca)".

Signatures	Date	% contribution
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Creator: <u></u>		<u>45%</u>
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Creator: <u></u>		<u>10%</u>
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Creator: <u></u>		<u>45%</u>
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5. **Uses and applications:**

*What are the immediate uses for the IP created and by whom?*

The IP created will be of immediate use to the producers of resins used for making composite materials, adhesives, sealants and other applications. These materials are in turn used by manufacturers of aircrafts, automobiles, sports equipment and by people making components for pipes, boats, reservoirs etc.

*Describe briefly future applications you foresee if there are any:*

Future applications can be epoxies used for adhesives, sports equipment such as bicycles, skis, golf clubs, sealants and many other structures.

6. **Development status:**

Documentation available.

Report on test and results available.

Prototype status:

Samples have been made and tested.

*What additional design changes have thought about?*

More samples need to be run to provide more confidence in the method and also to expand the range of applications.

**7. Disclosure/Publications:**

*List and attach copies of any articles or publications related to the created IP:*

There are many publications in the domain of polymer nanocomposites in the literature. However, the results described in these publications are not as good as the results obtained using the invented method described here. Most information can be found in the book "Pinnavaia T.J. and G.W. Beall, Polymer-Clay Nanocomposites, John Wiley & Sons, 2000". The applicants have sent a publication to be presented at the conference organized by the Society for the Advancement of Materials and Process Engineering (SAMPE) to be held in Long Beach, California in May 2003. In this publication, the benefits of the new development are described but not the method of processing itself. A copy of this publication is attached.

The applicants are of the opinion that Disclosure of the method of processing will be damaging to the process of obtaining IP rights.

**8. Market information:**

*What elements of your creation will provide a market advantage to the current state of the art?*

The great increase in fracture toughness of the material is the element that will provide a market advantage to the current state of the art. Fracture toughness is a very important properties of materials used to make aircraft structures. Having the fracture toughness increase by many times is a drastic improvement.

*Have you developed any industrial contact, interest or support? If so please list the companies with names of your contact.*

No. We hope that the presentation like the one at SAMPE will generate industrial interest and contact.

**9. Research support:**

*Provide details on the support provided by all sponsors for the research which led to the created IP.*

The main research support has been from the Discovery grants of the Natural Sciences and Engineering Research Council of Canada (NSERC).

*Identify if any of the funding support required or requires assignment of any IP rights associated with the created IP.*

The NSERC support did not require assignment of IP rights.

*List any person who have signed a waiver or ownership and attach copies.*

None.

**10. Work plan:**


*Identify the major avenues or phases of research, prototype development, patent and other work you foresee being required to facilitate the commercialization of the created IP:*

More tests need to be done to provide more confidence on the obtained results.

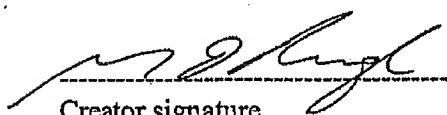
**11. Relationship with Gestion Valeo:**

*Indicate if you have met with Gestion Valeo representatives and if you have signed any agreement.*


No.

  
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Creator signature

  
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Creator signature

  
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Creator signature

## ASSIGNMENT OF OWNERSHIP RIGHTS

This form must be filled out by University Inventors who desires the University's assistance in pursuing intellectual property protection and/or technology transfer for a specified invention.

Inventors: Dr. Suong Van Hoa  
Mr. Weiping Liu  
Dr. Martin Pugh

Department: Mechanical and Industrial Engineering

Title of Invention: Process for the development of epoxy nanocomposites and products thereof.

Potential Sponsor(s): \_\_\_\_\_

*we* I, the inventors of the above device, product or process, hereby assign all right, title and interest in the Invention to Concordia University. I understand that I will be entitled to a share of any revenues generated by the Invention in accordance with the terms of the "Protocole d'entente sur la gestion de la propriété intellectuelle" herewithin attached.

Dr. Suong Van Hoa  
Inventor Name

*Suong Van Hoa*  
Signature

Mr Weiping Liu  
Inventor Name

*Lin Weiping*  
Signature

Dr. Martin Pugh  
Inventor Name

*M. Pugh*  
Signature

*Nabil Esmail*  
Nabil Esmail, Dean  
Faculty of Engineering and  
Computer Science